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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

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☐ *Additional inventors are being named on the ____ separately numbered sheets attached hereto*

TITLE OF THE INVENTION (280 characters max)

APPARATUS AND METHOD FOR QUANTATIVELY MEASURING LIQUID DRYING RATES ON SUBSTRATES

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Respectfully submitted,

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Date 08/08/02

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C-553 Patent Application

APPARATUS AND METHOD FOR QUANTATIVELY MEASURING LIQUID DRYING RATES ON SUBSTRATES

Field of the Invention

This invention relates to the determination and indirect measurement of liquids and liquid films. The invention also relates to apparatus and methods for determining the drying rate of a liquid film applied on a substance.

Background of the Invention

The need to determine the drying rate of a liquid film applied on a substrate frequently arises in a wide variety of contexts and industrial applications such as printing inks, paints, coatings, lacquers, etc. For instance, printing ink in its supply form is liquid (low viscosity liquids such as gravure or ink-jet inks or high viscosity liquids such as letterpress or lithographic inks) but it should change into solid form after application.

This change from liquid to solid state (drying) may occur in many ways:

1. Evaporation drying - the rate of drying depends on the evaporation rate of the solvents used,
2. Oxidation drying - the ink dries by oxidation as a result of chemical reaction between the ink and oxygen from the atmosphere,
3. Absorption drying - when ink and/or solvents can penetrate into the porous structure of the substrate,

4. Chemical drying - different than oxidation mechanism e.g. thermosetting inks or paints (chemical crosslinking takes place at the elevated temperature),
5. Radiation curing - polymerization of monomers and oligomers used in the ink or coating takes place when the liquid ink film is irradiated with ultraviolet (UV) light or electron beam (EB).

More details on the different mechanisms of drying can be found *e.g.* in "The Printing Ink Manual" by R. Leach.

Typically, liquids such as printing inks have been reported to dry on the basis of a simple scale having gradations ranging from "quick or fast drying" to "medium drying" and "slow drying". Determination of which of the three drying categories a particular liquid should be classified has usually been done on the basis of visual and/or tactile inspection. A sample of the liquid is applied wet to a substrate material, typically glass or metal, and the degree of dryness is visually or physically observed over certain pre-determined time periods. The liquid is observed over a range of time between several seconds to hours in duration, at which times the visual appearance of the sample is noted and/or the sample is touched in order to determine the tackiness of the sample. Thus, a sample is determined to be either dry or still drying and is correspondingly assigned either a "quick or fast drying", "medium drying", or "slow drying" designation. This method is extremely subjective and non-quantitative.

The appropriate drying rate of an ink may cause severe problems for printers if not known. For instance, during printing with gravure or flexographic inks ink may dry in the gravure cylinder or anilox roller cells. The dry ink can clog the cells of e.g. printing cylinder and make printing

impossible. Therefore, the drying rate of the ink is extremely important and has to be appropriately balanced to achieve the expected result e.g. high printing speed and high quality prints. Ink drying too fast may affect ink transfer in flexographic and gravure printing as the ink will dry either too fast or too slow on the plate, rollers or cells. Further printing problems include clogged print head nozzles and poor print quality in the final product e.g. ink-jet printing.

Another method used is a Geiger Press Test wherein the resolubility of a "dry" liquid is determined by applying ink onto the drying liquid film in a simulation of stopping and restarting a press and having the same printability as previous to the stoppage. If the "dried" film resolubilizes, the number of proofs necessary to get back to original state is noted and a drying classification and resolubility classification is determined for that liquid film. Again, this test is subjective and non-quantitative.

Moreover, it is extremely difficult or impossible to make any evaluation when liquid drying is very rapid. There are some instruments available on the market that allow for less-subjective evaluation of liquid drying such as PIRA ink drying time tester, IGT drying time recorder, and Thin Film Analyzer (TFA). These instruments are based on different principles and can be used only for high viscosity and relatively thick films (e.g. paints, coatings). They cannot be used to evaluate the drying rate of low viscosity, fast drying and thin films.

At the present there is no instrument capable of quantitative characterization of the drying rate of the liquids and liquid films. There is a need in the art for technique of measuring the drying rate of liquids and/or liquid films applied or to be applied on a substrate on a more precise, quantitative

basis. Because there is no simple way to directly measure the parameter "drying rate", it is, alternatively, necessary to measure some other physical property related to the liquidity or fluidity, that conversely relates to the degree of dryness of the liquid being measured.

The drying rate of the liquid may affect some experimental techniques used to characterize the properties of liquids such as e.g. dynamic surface tension (DST). The instruments that are used to measure the DST use the maximum bubble pressure (MBP) - BP2 tensiometer by Kruss, USA - or differential maximum bubble pressure (DMBP) - PC9000 by Sensadyne, USA - techniques and utilize single or two capillaries immersed in the liquid, respectively. Gas bubbles through the capillaries and the flow rate and the pressure are measured to calculate the DST of the liquid. Liquids that dry fast and have inadequate resolubility can dry inside the capillary and affect the DST (pressure) measurements.

While others have determined the surface tension of a liquid, no one has associated the DST (pressure) measurements with the drying rate of liquids and proposed this principle to quantify the drying rate of liquids. The DST of liquids can be measured with relative ease. U.S. Patent No. 3,881,344 entitled "Monitor for Continuously Measuring Surface Tension of Liquids" discloses an apparatus for measuring the DST of a liquid in a flowing stream, whereby a capillary tube is dipped in the liquid and a bubble is formed on the end of the tube by forcing air down the tube. The pressure required to form the bubble at the capillary tip below the liquid surface is related to the surface tension of the liquid via the following expression:

$$= (d \quad P) / 4$$

where is the dynamic surface tension in dynes/cm or mN/m;

d is the effective diameter of the capillary tube in cm; and

P is the excess of the inert gas pressure required to generate bubbles at the capillary tip immersed in the liquid at a given rate, in dynes/cm^2.

However, the association between this pressure (or the DST of the liquid) and the drying rate of that liquid was not explored and developed.

Moreover, there exists a need to be able to quantatively measure the drying rate of a wet liquid by virtue of monitoring the changes in electrical conductance and resistance associated with the liquid as it dries. No one has developed a way to measure the drying rate of such liquids via electrical conductance/resistance until now.

Summary of the Invention

Accordingly, the invention is an apparatus and method for determining the drying rate of a liquid applied on a substrate which provides quantitative measurement correlated with the drying rate of liquids applied on the substrate.

According to another embodiment of the invention, there is provided an apparatus and method for measuring DST pressure as related to the drying rate of a liquid and/or liquid film.

According to another embodiment of the invention, there is provided a method for quantative measurement of liquids based on the print density of a liquid at certain intervals after roll out on a substrate

According to another embodiment of the invention there is provided an apparatus and method for measuring directly the

electrical conductance or inversely the electrical resistance of a liquid film and using these measurements as an indirect measure of the drying rate of the liquid film.

Brief Description of the Drawings

Figure 1 is an overall view of the roll out and calculated drying rate of a liquid by measuring the density of the liquid after application to a substrate.

Figure 2 is an overall view of the apparatus for measuring the drying rate of a liquid by measuring the pressure required to generate bubbles of gas at the capillary tip immersed in the liquid.

Figure 3 is an overall view of an apparatus for measuring the drying rate of a liquid via the electrical conductance (resistance) measurements of the liquid applied to a substrate.

Detailed Description of the Invention

The invention of quantatively measuring the drying rate of a liquid applied to a substrate is described herein in four methods and three corresponding apparatus for measuring the drying rate of a liquid applied to substrate.

A method is disclosed wherein the drying rate a liquid applied to a substrate is quantatively measured by the print density of the liquid at certain intervals of the liquid being applied onto the substrate.

Generally, according to the method, a controlled volume of the liquid to be tested is applied to an application means such as a handproofer and is rolled-out on a substrate material, to

form an initial (I) proof. The ink on the application means is allowed to dry over a pre-determined period of time, then re-applied to the application means and rolled-out on a fresh substrate, to form the after (A) proof. A densitometer is used to read the end of each "tail" of the rollouts providing a print density or density number. The tail is the visible change in density of the liquid on the substrate as it is being rolled out on the substrate. The tail is then read by a densitometer in order to determine the density of each separate tail.

The density reading data obtained from the rollouts is used to determine the drying time of the liquid, which is calculated based on the number of tails and the density of each tail. The density reading data is then used to plot a curve, for the I proof and the A proof and subsequent I and A curves are then graphed. The area between the curves is then calculated and the coefficients of an empirical model are determined.

The method includes loading a liquid in a set volume, which can be any volume, as long as repeated throughout the method, onto an application means. For tests of drying rate of an ink on a substrate, the ink should be tested at its press viscosity. The liquid is discharged through a disposable pipet tip and one pipet should be used per rollout and not reused. As see in **Figure 1,** the liquid film is applied to a substrate via any conventional application apparatus including a flexo press or handproofer, such as an Early Enterprises Handproofer. An initial (I) rollout from the application apparatus is made onto the substrate utilizing a constant angle, pressure and speed of application. The substrate may be made of paper or film. Suitable substrates include paper, plastics, glass, film or the like. For tests on film substrate, a film such as AET Films T 523-3 is preferred. As necessary according to the liquid

applied, film samples are oven dried at a temperature of around 80 °C for the time necessary to cure. After sufficient time, preferably around 5 minutes, for the liquid to dry on the application apparatus, the liquid is once again applied to the application apparatus in the same volume and rolled out on the same type of substrate via a new pipet tip, again using constant angle, pressure and speed. This is referred to as the after (A) rollout.

The overall method is repeated as many times as necessary in order to arrive at an average graph or model fit through data analysis.

The tail of each rollout is located and marked with a circle for scanning into a densitometer. Multiple repetitions of the I and A rollouts are preferably performed so that the average values and/or model fit can be determined.

Once the average and/or model fit density of each tail is calculated, the average and/or model fit density is plotted on a graph versus the tail number for both the I rollout and the A rollout. The slope of curve of the I and A rollouts are referred to as the I drying proof curve and the A drying and resolubility proof curve. The I curve represents drying, transfer and absorption. The A curve represents drying, transfer, absorption and resolubility. The area between the I and A curves represent both the resolubility and the drying rate of the liquid.

Generally, it was found that faster drying liquids had less tails and a greater slope of the curves. Correspondingly, slower drying liquids had more tails and a smaller slope of the curves.

The method allows liquids to be distinguished on the basis of their drying rate and resolubility. The method may also be

used to test the multiple (typically four or more) colors of a process ink set to determine whether all colors print process equally, dry at the same rate and therefore set together o as to determine if different diluting solvents are required for the inks of the same process set to have good printability. Thus, the method can also be used to differentiate between liquid inks for drying and resolubility to determine if cutting solvent mixture needs to be changed for a particular pigment. The method can also be applied to traps and used to predict the tracking and trapping of the liquid in question. Tracking is noted on a substrate due to poor drying while smearing on the substrate might be due to poor resolubility. One of the reason for poor trapping may be poor drying and poor resolubility wherein one ink dries either too fast or too slow before the trapped ink is applied and can result in increased costs to the press when it occurs. The method may also be used to distinguish between process and non-process printers in that the process printers must be slow drying in order to reproduce the print required in sufficient clarity. For example, differences have been observed between magenta and cyan color inks within the same base system.

A further apparatus and method for measuring the drying rate of a liquid is disclosed using the discovered relation between the pressure (or the apparent DST of the liquid) necessary to generate a bubble at the capillary tip immersed in a liquid, as the liquid dries and therefore clogs the capillary reducing the radius of the capillary, and the related drying rate of said liquid.

Surface tension is commonly measured using static, equilibrium techniques such as the duNouy tensiometer. DST measurement thus frequently uses the maximum bubble pressure

method (single capillary) or the DMBP method (two capillaries of different orifices). Gas passes through an orifice tube into a liquid, forming a succession of bubbles. The inflation pressure inside each bubble is at a maximum when the bubble achieves minimum radius of curvature equal to the capillary as established in Equation (1) above. The maximum bubble pressure occurs as the bubble assumes hemispherical shape at the orifice. The maximum bubble pressure is thus directly related to, and provides a true measure of, the surface tension of the liquid. (W. J. Moore, Physical Chemistry, 3rd Ed., p. 729-31, Prentice Hall, N.J., 1962.) Using these instruments, maximum bubble pressure is observed as a value averaged over a number of bubbles generated at a fixed rate. When more than one rate is to be studied, the rate of bubbling must be changed manually or automatically, and another maximum pressure average value taken.

It has been discovered that the drying rate of a liquid can be characterized quantitatively by measuring the increased pressure (or resulting apparent DST of a liquid). This measurement is based on the observation that the liquid may dry in the capillary tube and thus decrease the effective radius of the capillary tube, thereby increasing the pressure required to generate the bubbles. This increased pressure and related apparent DST are correlated to the drying rate of the liquid.

Thus, we have discovered that using the same principle as that used for the DST measurements one can obtain the qualitative information about liquid drying rate. To do so one can design a new instrument capable of accurate measurements of the pressure at the capillary tip or use conventional DST tensiometers to monitor the pressure necessary to generate bubbles in a liquid through a capillary tube tip immersed in the liquid. At the beginning of the method, liquid enters the

capillary (upward) and wets the inner capillary wall. The increasing gas pressure in the system gradually expels the liquid from the capillary and finally a bubble is formed at the capillary tip. When the liquid flows downward in the capillary a thin liquid film is deposited on the inner capillary wall. The thickness of the film in the capillary depends on the liquid properties e.g. viscosity. The gas flowing through the capillary is generally very dry as a necessary requirement due to the electronic system and pneumatic system elements that are sensitive for any condensation, e.g. water inside the instrument, such that the liquid film on the inner capillary wall will dry until it is re-wetted with another portion of the liquid that will re-enter capillary when the pressure in the capillary drops due to bubble release. During the DST measurements this cycle will be continuously repeated. For the liquids that are not perfectly non-drying or their dry films are not perfectly re-soluble, gradual clogging of the capillary can be visually observed and quantatively observed through the increased pressure needed in order for the bubble to form in the smaller diameter capillary. The presence of deposit on the inner capillary wall will reduce the effective capillary radius and according to Equation (1) a higher pressure will be required to generate bubbles at the capillary tip. This phenomenon is schematically illustrated in the Examples 7-8. It has been discovered that the extent of the capillary clogging by the drying liquid depends on the drying/resolubility properties of the liquid. Therefore, this principle can be used to quantify the drying rate of the liquid, e.g. liquid printing inks.

Using the capillary clogging principle to characterize the drying/resolubility properties of the liquid one can measure the net effect. Generally, it was found that faster drying

liquids had a greater slope of the curve of pressure needed in order to generate bubbles. Correspondingly, slower drying liquids had a smaller slope of the curve of pressure needed in order to generate bubbles.

According to **Figure 2**, a single capillary apparatus for measuring the drying rate of a liquid as related to its apparent dynamic surface tension is described. A reservoir or container **20** for holding a volume of liquid **30**, said volume not being critical and generally between about 20 to about 100 ml, although smaller or larger volumes may also be utilized, is provided. In order to meet the requirements of the apparatus, the volume of liquid **30** must be sufficient so that the formation of gas bubbles **50** in the liquid can readily be detected. The shape and materials of the liquid reservoir or container **20** is not critical. Typically, an open cylindrical glass or plastic beaker may be utilized. The material of the container **20** should be inert with respect to the liquid **30** whose drying rate is being determined. Glass, polyethylene, or polypropylene vessels are typically adequate for use with most liquids. If the liquid **30** has extreme pH values, typically either less than about 3 or greater than about 11, or is otherwise corrosive, a resistant material should be used for the material of the container **20**. It is preferable that the material be translucent rather than opaque in order to facilitate observation of gas bubble formation in the volume of liquid **30** in the container **20**.

To function as part of the apparatus, it is only essential that the capillary tube **40** be capable of being inserted into the liquid. The capillary tube **40**, typically a cylindrical tube, of known inside diameter, is open at both ends. The capillary tube **40** has a first open end **60** for insertion into the volume of liquid **30** whose drying rate is being measured, present in the

above-described container **20**, and a second open end **70**, to which a supply source **80** of an inert gas is capable of being attached. The capillary tube **40** may be made of any material that is inert to the liquid **30** whose drying rate is being measured. The material of the capillary tube **40** should be such that generally it is liquiphilic and it is not liquiphobic to the liquid **30** whose drying rate is being determined, so that the liquid **30** is capable of wetting the material of the capillary tube **40**. The capillary tube **40** is typically glass, metal or plastic, and may be of any material that is not reactive with the liquid **30** or the inert gas **80**. The length of the capillary tube **40** is not critical, and must only be sufficiently long to be able to draw a sample of the liquid **30** from the container **20** into the capillary tube **40** through its first open end **60** that is immersed into the volume of the liquid **30** in the container **20**, and to be able to attach the supply source **80** of inert gas to the second, opposite open end **70** of the portion of the capillary tube **40** that remains above the liquid **30** in the container **20**, in order to be able to direct a stream of inert gas into the capillary tube **40** downwardly and countercurrently to the upward direction in which the liquid **30** is drawn from the container **20** into the capillary tube **40**. Typically, the capillary tube **40** has a length of from about 2 inches (about 5 cm) to about 4 inches (about 10 cm).

More than one capillary tube may be used and inserted into the liquid **30** e.g. Sensadyne PC9000 tensiometer that uses the DMBP principle has two capillaries of different orifices. If more than one capillary tube is used, then the pressure difference between the capillaries of different diameters is measured and taken into calculation of the DST.

The inert gas must not be chemically reactive with the

liquid **30** whose drying rate is being measured. The inert gas should be capable of being delivered at a variable pressure. The pressure and the volume of the gas being delivered, should be capable of being accurately measured. The inert gas utilized can be any gas that is inert to and not chemically reactive with the liquid whose drying rate is being measured. Typically, dry nitrogen is used, although other inert gases, including the noble gases, such as helium and argon may be used. This apparatus is used for determining the pressure (or the dynamic surface tension of the liquid **30)** and accordingly the drying rate of the liquid **30** as set forth in the method below.

According to a method for determining the drying rate of a liquid utilizing the above-described apparatus, a volume of the liquid whose drying rate is to be determined is poured into a container. An inert gas supply source is then connected to the second open end of a clean capillary tube of known inside diameter and radius. The capillary tube is then positioned in the container of liquid such that the first open end of the capillary tube is immersed below the level of the liquid in the container in order for liquid to be drawn from the volume of liquid in the container into the capillary tube by the hydrostatic pressure and capillary action. It is preferred that the first open end of the capillary tubes be completely immersed below the liquid level in the container and the depth of immersion to be kept constant when comparing different samples.

The pressure of the inert gas should be noted. This pressure may increase upon time as the liquid may dry inside the capillary tube. During each cycle (generation of single bubble at the capillary tip) at first the liquid flows into the capillary (upward) due to capillary action and the hydrostatic pressure. After some time the liquid flow upward is stopped by

the increasing gas pressure in the system. Further increasing gas pressure subsequently expels the liquid from the capillary (flow downward). At a certain time the pressure reaches the maximum value when the liquid meniscus curvature is equal to the radius of the capillary. Further pressure increase in the system causes instant bubble burst and release from the capillary. This causes that the pressure in the system drops and liquid re-enters the capillary upwards. Liquid exiting the capillary leaves the thin liquid film on the inner capillary wall. This film will dry (drying time) until it is re-wetted by the liquid re-entering the capillary and moving upward after bubble release (resolubilization time). The volatile matter from the liquid will be removed from the system by the stream of gas forming bubbles. If the semi-dry film is completely resolubilized during resolubilization time then no net deposit will be observed inside the capillary. All liquids other than perfectly non-drying or whose dry films are perfectly resoluble will gradually clog the capillary upon time. The rate of gas pressure increase in the system will depend on the extent of this clogging. Therefore monitoring this pressure can deliver information about drying properties of the liquid.

From the foregoing, it is evident that the apparatus and method of measurement for determining the drying rate of a liquid via the pressure measurements that is required to generate bubbles at a constant flow rate (or its apparent dynamic surface tension) are for use in the ambient atmosphere. It is to be understood that it is possible to measure the drying rate of a liquid under a different atmosphere by providing a different atmosphere in the capillary tube under which the liquid sample is drawn into the capillary tube. Thus, the temperature and/or e.g. humidity of the gas in the capillary

tube can be controlled to provide such a different atmosphere. It is also possible to provide air or a completely different atmosphere than air by providing a different gas in the capillary tube. Either of these can be accomplished in a number of ways, such as by placing the entire apparatus, including the capillary tube, in an enclosed hood into which air of a controlled temperature and humidity is introduced, or for a different, non-air atmosphere, by evacuating the air and introducing a different gaseous atmosphere of controlled and measured temperature and humidity.

Another apparatus and method to measure the drying rate of a liquid is disclosed by measuring the electrical resistance (or electrical conductance) of liquid films. Electrical conductance (EC) of wet film is much higher than the EC of the dry film. Similarly, the electrical resistance (ER) of the wet film is much lower than the ER of dry film. Therefore, this technique can be applied to quantify the drying rate of any system that has different EC (ER) in the wet and dry states without any limitations based on the liquid film's thickness, viscosity or drying speed.

The apparatus **100** of Figure 3, includes a pair of first **110** and second **120** strip electrodes that are disposed on a surface of a non-conductive substrate material **130** in a flat, co-planar manner, oriented in a generally parallel disposition with respect to one another. The set of electrodes **110, 120,** can be made of different materials such as metal or conductive polymer, metal being preferred. The set of electrodes, **110, 120,** can be placed in different lengths apart from one another and may be of different lengths themselves. The electrodes are attached to a means of measuring electrical conductance and/or resistance **140,** such as an ohmmeter, resistivity meter or conductivity meter.

The substrate **130** may be formed of Leneta Board, plastic, glass or any other nonconductive substrate material that does not affect the electrical conductance of the liquid film being applied onto the substrate **130**.

The method of determining the drying rate of a liquid film by measuring the liquid film's electrical conductance/resistance includes applying the liquid film **150** onto the substrate, by use of application apparatus **160** which may be a handproofer or Mayer bar or any other way to apply the liquid film **150** onto the substrate. Other forms of application of the liquid film **150** may be employed. The method utilizing the apparatus of Figure 3 can be used for a wide variety of materials, including all types of printing inks, paints, coatings, varnishes, lacquers, adhesives and the like. The method is well suited for use in testing gravure and flexographic inks, including water-based and solvent-based for paper packaging, solvent-based for film, and water-based for film. The method can be used for any system with different electrical conductance or resistance in wet and then dry states - no limitations regarding sample viscosity, ink film thickness, drying speed or chemistry.

According to the method for determining the drying rate of a liquid from measurement of either its electrical conductance, or inversely, its electrical resistance, involves the preparation of a rollout sample of the liquid on a substrate. The liquid film is applied to a substrate having at least two electrodes attached to a resistivity (conductivity) meter. The liquid film's electrical conductance is measured over a period of time. Wet liquid film has a relatively high electrical conductance ad it decreases as the liquid film dries. Thus, the drying rate may be calculated based on the decrease in the liquid films electrical conductance. Correspondingly, the

electrical resistance of wet liquid film is relatively low and increases as the liquid film dries. Again, the drying rate may be calculated based on the increase in the liquid film's electrical resistance. Graphs should be plotted monitoring the electrical resistance versus time and the electrical conductance versus time to obtain a characteristic determination.

From the foregoing, it is evident that the apparatus and method of measurement for determining the drying rate of a liquid film via the electrical conductance (resistance) measurements of said film are for use in the ambient atmosphere. It is to be understood that it is possible to measure the drying rate of a liquid under different experimental conditions (atmosphere, temperature, humidity etc.). Thus, the temperature and/or e.g. humidity can be controlled e.g. in a measuring chamber. It is also possible to provide air or a completely different atmosphere than air by providing a different gas. Either of these conditions can be accomplished in a number of ways.

The following examples illustrate the invention. The examples use multiple colors (cyan and magenta) of fast, medium and slow drying systems and said systems are applied on many substrates, including both a porous paper substrate and a non-porous substrates, using a conventional means.

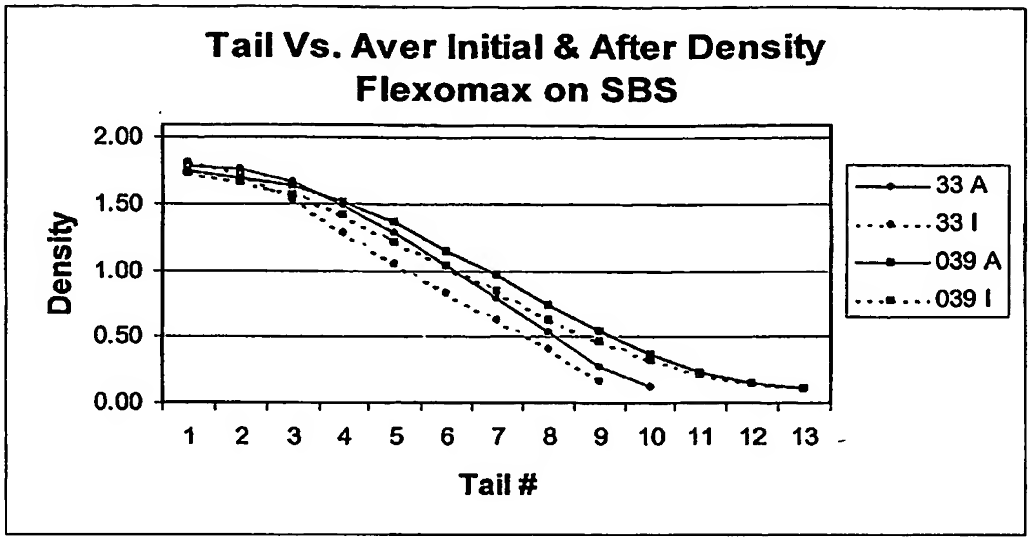
Example 1

Flexomax® 33 (fast drying) and Flexomax® 039 (slow drying) cyan inks were measured separately to determine their respective drying rates. Each ink was applied to an Early Enterprises® 600 (2.8 BCM) handproofer with a volume of 2.8 BCM and rolled out onto a separate solid bleached sulfite paper liner ("SBS") and

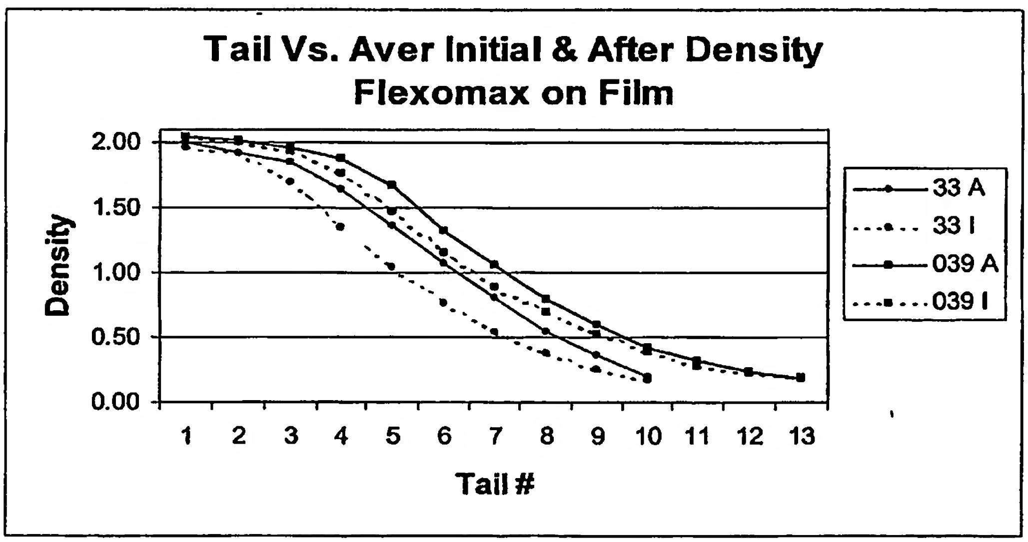
polypropylene film (AET Films®, T 523-3) via a disposable pipettes tip (Eppendorf pipet with 30 µl capacity). The rollout was made using constant angle, pressure and speed and the proof was heated for 10 seconds at 80°C to dry the ink film. The first rollout was designated as Initial Rollout ("I"). After five minutes had passed, the ink was reapplied onto the handproofer which contained some of the dried ink from the first application of the respective ink, and rolled out again for each ink on the substrate. The prints were heated as necessary in order to dry on the respective substrate. The second rollout was designated as After Rollout ("A"). The process was duplicated twice again on new substrates via new disposable Eppendorf pipet tips, in order to arrive at an average "I" and "A" for when the results were graphically plotted.

The inks left visual tails on each substrate. The tails of each ink in each of the three I and A rollouts were read with an X-Rite® 428 Densitometer, to obtain the print density of each tail. The average density of each tail for the three I and A rollouts was then plotted on a graph. The joining of each plotted density point on the graph formed a before (I) and after (A) drying proof curve, giving a fingerprint of the ink.

The inks were thus evaluated by comparing the respective drying profiles for each ink, which consist of a before drying proof curve and an after drying proof curve for each ink. Differences between the before and after drying proof curves are graphed to provide curve shapes. The parameters of the curves under comparison include differences in the slopes of the before and after drying proof curves for the two inks, the overall shapes of the before and after drying proof curves, and the area between the before and after drying proof curves. The graphs as plotted are as follows in Graph Nos. 1 and 2:



Graph 1



Graph 2

The I curve represents the drying, transfer, and absorption of the ink. The A curve represents the drying, transfer, absorption, and resolubility of the ink. The number of tails represents the drying. The area between the A and I curves, the

numbers of tails, the coefficients, and the curve shape and slope can be used to differentiate between the ink systems.

Observations drawn from the graphs are that the greater the number of tails, the slower the drying. Further, faster drying inks tended to have curves of greater slope than that for slower drying inks. Also, the curves for the faster drying inks tended to have a shorter tail and exhibit a greater area between the before and after drying proof curves. The overall area between the curves was greater for polymeric (e.g. polypropylene film) type substrates than for paper nitrocellulose based (e.g. solid bleached sulfite paper) type substrates, indicating less absorbency of the ink into the polymeric substrate. The area between the before and after drying curves for each ink was calculated and is shown in the following Table.

Table 1

INK	SUBSTRATE	AREA
Flexomax® 33	SBS	1.25
Flexomax® 039	SBS	0.82
Flexomax® 33	Film	1.68
Flexomax® 039	Film	0.91

The results of this test were verified as feasible on water-based systems by performing another test on UltraCorr GCMI® 74 Red Ink on SBS.

Example 2

Using the same equipment and same process as in Example 1, Sun Chemical® XV-98 33 magenta ink and 39 cyan ink were tested. And also Flexomax® 33 magenta ink and 039 cyan ink, were each

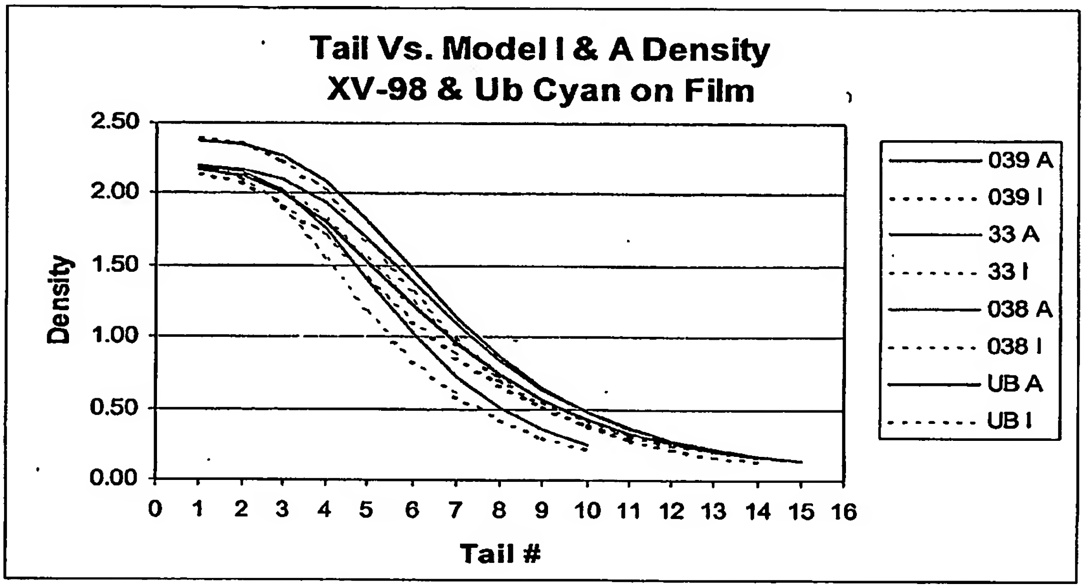
tested on polypropylene film. The drying time between initial (I) and after (A) rollout was optimized so that the time between tests was five minutes for optimal results. For both systems (the Sun Chemical® XV-98 and Flexomax® inks) the magenta 33 ink had fewer tails, a greater slope and greater area between curves and therefore was observed as a faster drying ink. Using a Grind Gauge test for drying, it was determined that the Sun Chemical® XV-98 33 ink was the fastest drying ink and using a Geiger press to simulate ink resolubility, this same ink showed the least resolubility. The Sun Chemical® XV-98 33 ink demonstrated greater overall area between the curve, which was likely attributed to faster drying into the cells giving a greater slope, lower density and a greater amount of the ink drying into the cells. When the Sun Chemical® XV-98 33 ink was deposited for the A rollout, it resolubilized a greater amount of dried ink, giving a higher A curve. The area between the before and after drying curves for each ink was calculated and is shown in the following Table. This area corresponds to the resolubility and drying rate in the much faster drying 33 inks had a higher value than the 39 inks due to a greater amount of drying as shown in the cells of the I curve.

Table 2

INK		AREA
Flexomax®	Sun Chemical® XV-98	
33	-	1.61
039	-	0.72
-	33	1.08
-	039	0.67

Example 3

Using the same equipment and same process as in Example 1, Sun Chemical® XV-98 33, Flexomax® 039, Flexomax® 038, and Ultrabond® cyan inks were each tested on polypropylene film. A model curve was fit to the raw density data of each tail versus tail number and was graphed as set forth below in Graph No. 3:



Differences and trends were noted with respect to the base system. The model curves for the Sun Chemical® XV-98 33 ink had fewer tails, greater slopes and greater areas and thus was faster drying. The difference graph below shows four ink systems comprised of a colorant base and let down varnish -- Sun Chemical® XV-98, Flexomax®, Sunfine® and Ultrabond®. The first three systems were shown with three base systems (33, 038, and 039). All systems demonstrated similar shapes with the bases. The fast drying 33 inks are not process printers in that the inks need not be slow drying for reproduction on a substrate. The magenta inks were then also tested and produced comparable

results as with the cyan inks.

The results for area between the curves are shown in the following Table 3, wherein area corresponds to the drying rate and resolubility for the cyan inks:

Table 3

INK	AREA
XV-98, 33	1.14
XV-98, 038	0.95
XV-098, 039	0.77
Ultrabond	1.07

Again, the 33 ink showed a greater area due to the much faster drying and higher ink deposit in the cells for the I curve.

Example 4

Using the same equipment and same process as in Example 1, water-based flexographic packaging inks were applied at a greater volume 3.7 BCM and were tested for differences which would lead to tracking or poor trapping. Two cyan inks were tested, one with a low pH of 8.48 and the other with a high pH of 9.60. Differences were noted between cyan inks as a result of the pH of the cyan ink. The low pH cyan ink exhibited an area of 1.09 while the high pH cyan ink exhibited an area of 3.30. The high pH cyan ink therefore had a greater resolubility (area) and comparable drying rate versus the low pH cyan ink. These results were independently confirmed using other methods (i.e. grind gauge and electrical resistance) and the difference in volume from earlier tests did not affect the final results of the drying rate.

Example 5

Using the same equipment and same process as in Example 4, cyan inks containing different resins were evaluated to determine the effect of the resin on resolubility. Cyan ink was made by combination with three different resins (A -styrene acrylic 2, B -fumerated rosin ester, and H - styrene acrylic 1) and then each system was applied separately by the handproofer on T 523-3 substrate (polypropylene film). The 3 inks ere tested in a tr5ap with the white in as the second down ink. The experiment was repeated with each cyan ink. The drying speeds of the three cyan inks were similar.

The resolubility of ink H by the white ink was much greater than that for ink B, which was greater than that for ink A. The high pH cyan ink of Example 4 was a remake of H.

For Examples 4 and 5, drying rate differences between inks of the same ink formula were attributable to the instability of the ink. The results for area between the curves, representing the drying and resolubility rate, are shown in the following Table 4:

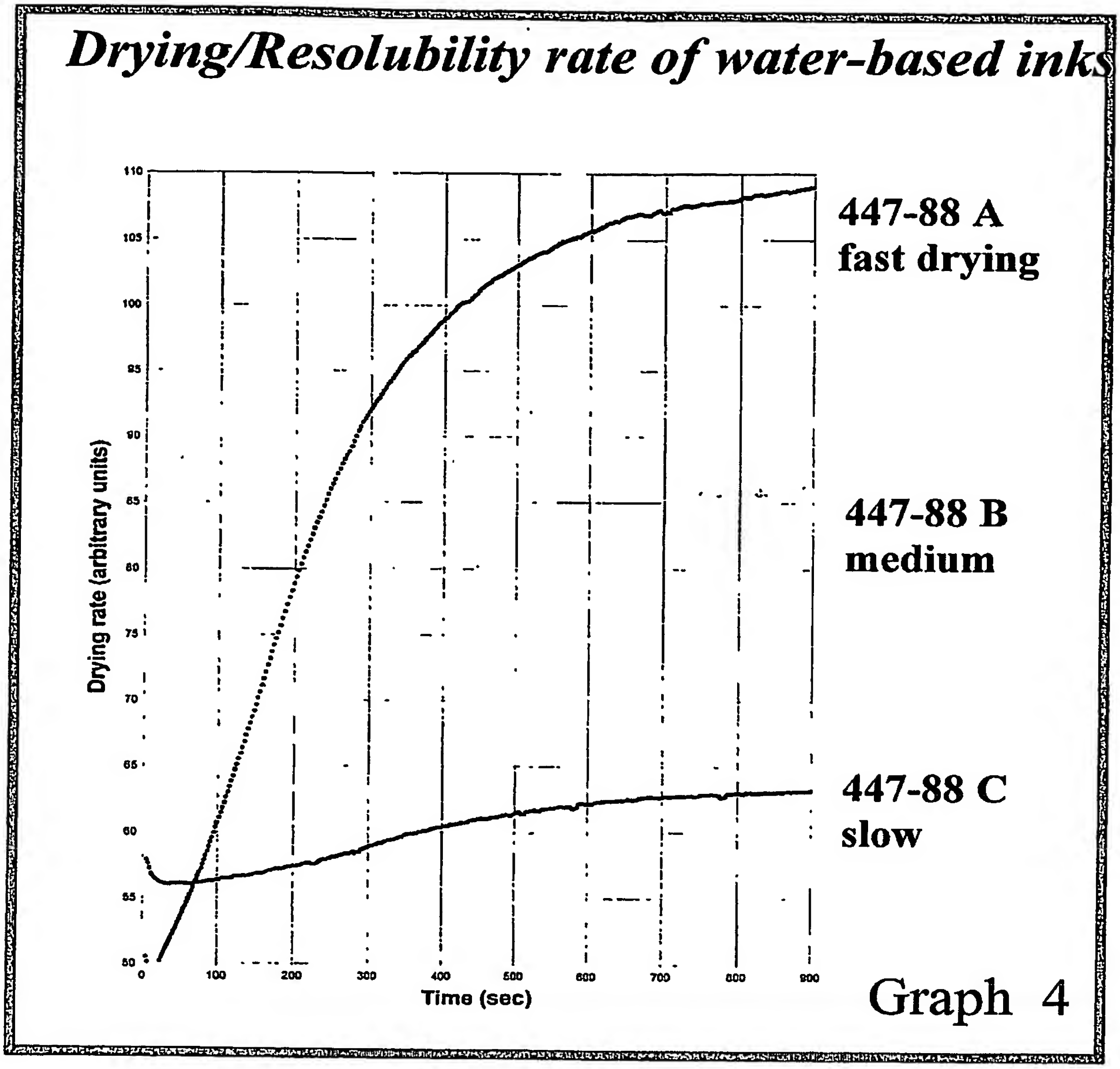
Table 4

INK	AREA
A	0.77
B	1.96
H	3.54

As can be seen, the above method allows for quantification of the drying rate of liquid flexographic inks. The reproducibility of the results obtained by the method were independently confined in a separate experiment which yielded very good matching results.

Example 6

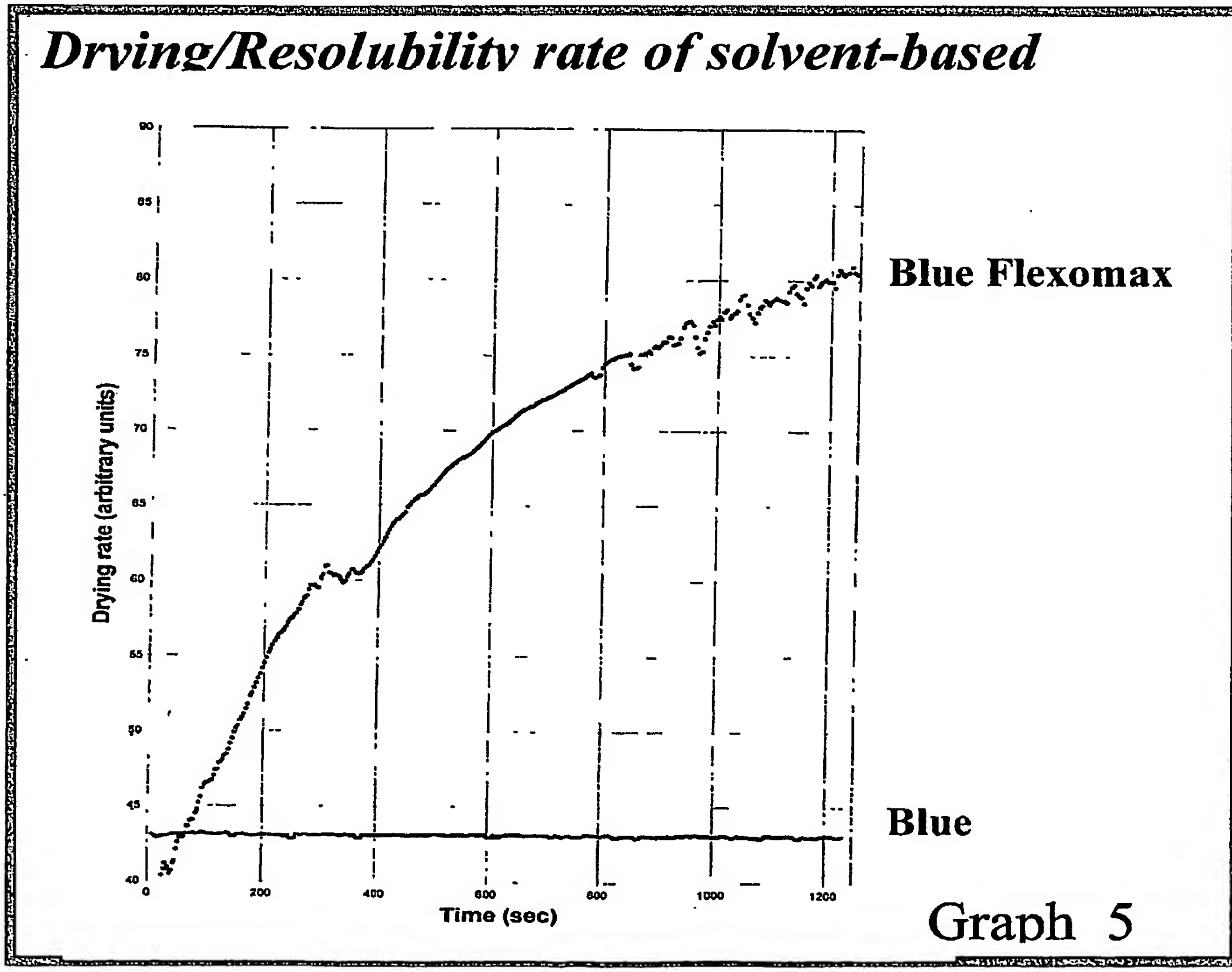
Examples 6, 7, and 8 refer to the apparatus presented in Fig. 2. For Example 6, three different samples of water based flexographic inks (Flexographic Packaging Red 477-88) carrying a manufacturer's rating of either being "fast", "medium" or "slow" drying were placed in a container through which dry nitrogen was bubbled via a stainless steel capillary (0.5 mm in diameter). The pressure and the resulting apparent dynamic surface tension (DST) versus time of each ink was then noted using a Sensadyne® PC500L instrument (available from Sensadyne, Mesa, AZ and equipped with two stainless steel probes, one having a large orifice - 4 mm in diameter - and the other having a small orifice - 0.5mm in diameter). The instrument was calibrated using water and ethyl alcohol and the initial bubble rate was about 3 bubbles/second. The apparent DST for each ink was measured over 15 minutes respectively. The apparent DST value increased over time, due to ink drying inside the capillary, until it reached a plateau or steady state value. The difference between the initial and final apparent DST value represents the extent of ink drying and the slope of the curve represents the drying rate. The apparent DST values over the time period obtained for all three inks are presented in Graph No. 4 below:



As seen, the graph clearly shows different "drying/resolubility profiles" for the inks drying at different rates.

Example 7

Using the same equipment and same process as in Example 6, solvent based laminating ink Flexomax® 33, carrying a manufacturer's rating of being a "fast" drying ink and Flexomax® 039, carrying a manufacturer's rating of being a "slow" drying ink were placed in the container and tested via their respective DST. The results obtained after measurements over a period of 20 minutes are shown in graph No. 5 below:

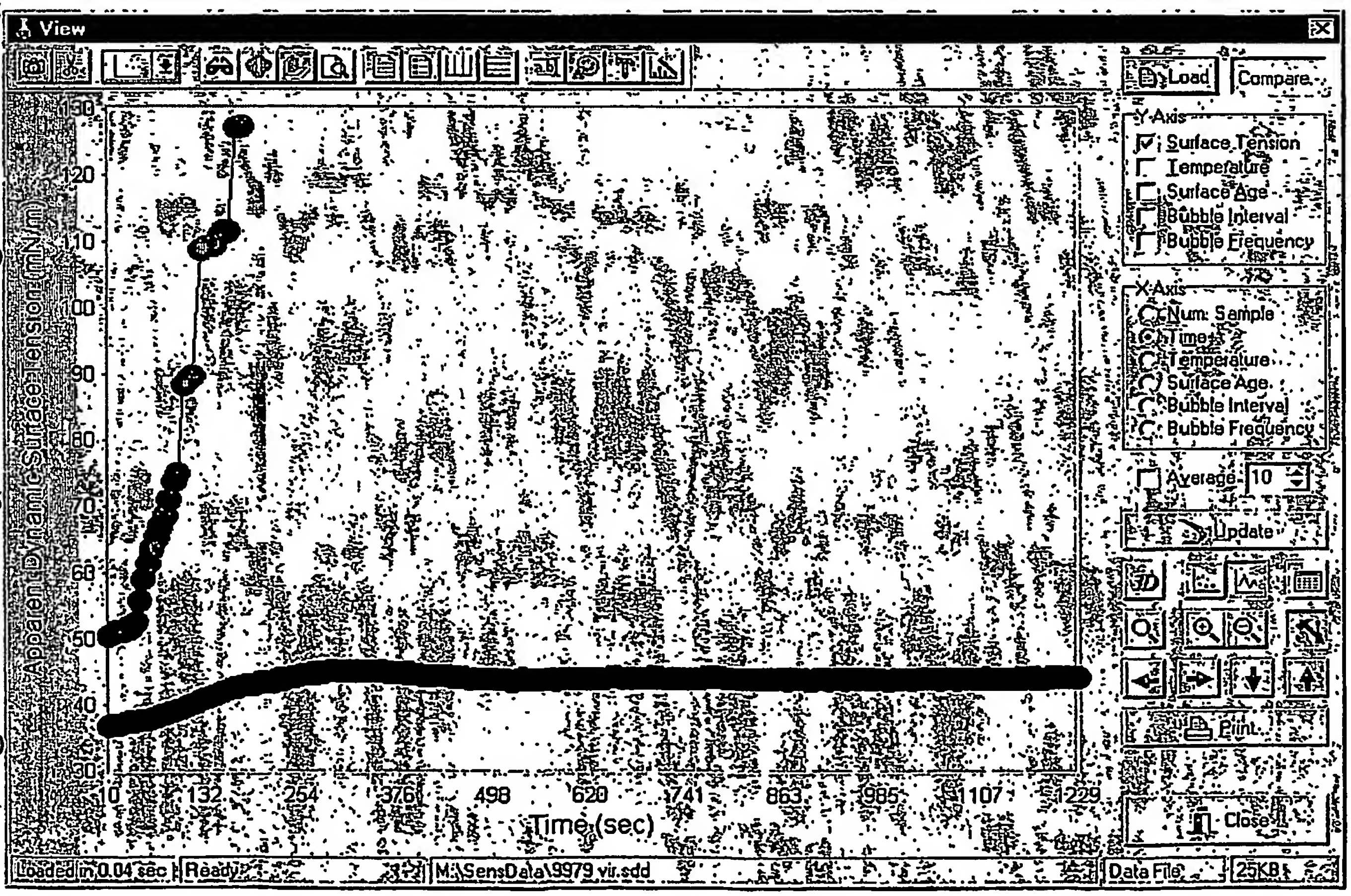


Graph 5

As can be seen, the above method allows for quantification of drying of liquid inks. The reproducibility of the results obtained by the method was independently confirmed in a separate experiment, which yielded very good matching results.

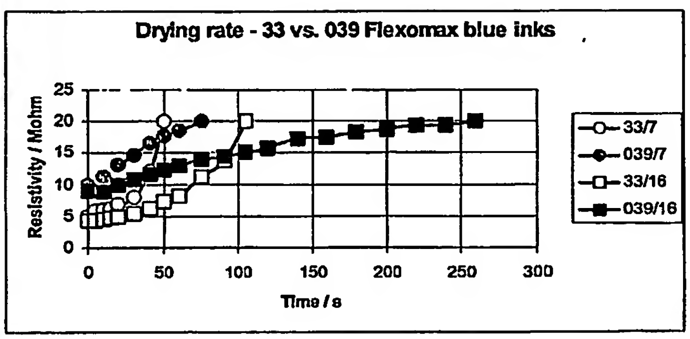
Example 8

Using the same equipment and same process as described above in Example 6, two toluene based publication gravure inks (one containing high level of solids and the other diluted with toluene) were tested for their respective apparent DST to profile their drying rate. The results over a measurement of 20 minutes are shown in Graph 6 below.



Example 9

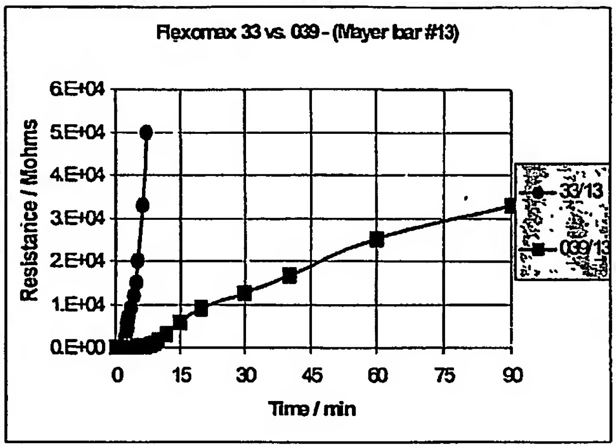
Examples 9 through 17 refer to the apparatus presented in Figure 3. Solvent based laminating inks, Flexomax ®33 (fast drying) and Flexomax® 039 (slow drying), were applied via Mayer bars #7 (33/7 and 039/7 - thin film) and #16 (33/16 and 039/16 - thick film) onto a Leneta Board ™ having an imprinted electrodes on the surface of the Leneta Board™. The length of each pair of electrodes was 7 cm and the space between the electrodes was 0.3 cm. Changes in the inks film resistance was recorded using a Radioshack® multimeter (having a resistance range of 0-20 Mohm). The results obtained over a period of 5 minutes are presented in Graph No. 7 below:



Graph 7

<u>Example 10</u>

Using the same inks as in Example 9, one bar was used, Mayer Bar #13 to apply the inks stated above onto a Leneta Board ™ having a pair of electrodes imprinted which were 20 cm each and spaced 0.5 cm apart. The changes in ink film resistance were measured using a Fluke® 189 digital multimeter. The results obtained over a period of 90 minutes are presented in Graph No. 8 below:

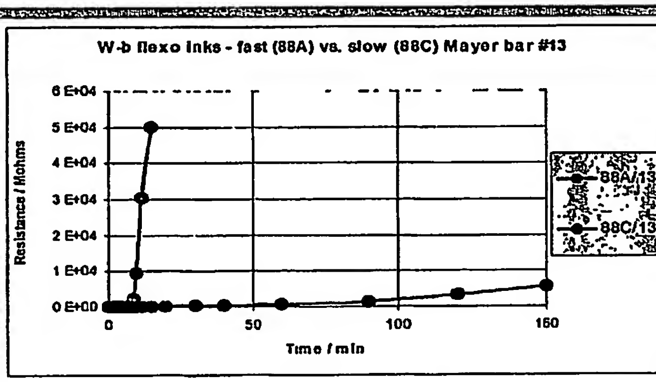


Graph 8

As can be seen, the resistance depends on the drying rate of the ink. For a constant ink film thickness and under similar experimental conditions, film resistance over time for a given liquid will be a unique curve having a shape influenced primarily by the drying rate of the ink.

Example 11

Using the same equipment and same process as in Example 9, water based flexographic ink, Sun Chemical® Flexographic Packaging Red 477-88, carrying a manufacturer's rating of either being "fast" (A) or "slow" (C) drying, was applied on the substrate via Mayer Bar #13. The changes in the ink film's resistance were measured using a computer inter-faced, Fluke® 189 digital multimeter. The results obtained over a period of 3 hours are presented in Graph No. 9 below:

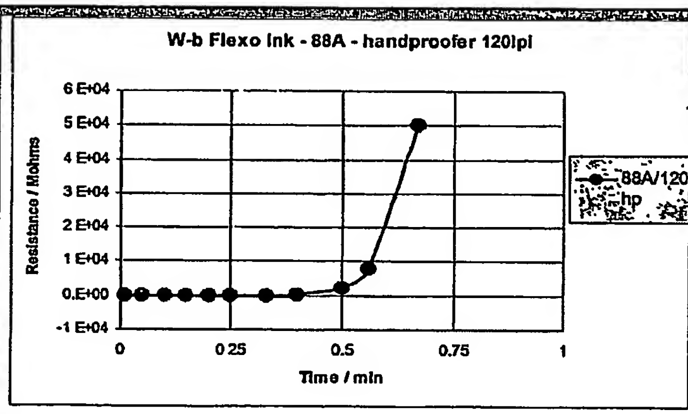


Graph 9

As can be seen, the quantification method correlated well with the manufacturer's drying rate profile for each ink.

Example 12

Using the same ink as in Example 11, the ink was applied to the Leneta Board ™ of Example 9, via a 120 lines per inch flexographic printing Early Enterprises® handproofer. The changes in the ink film's resistance were measured using a computer inter-faced, Fluke® 189 digital multimeter. The results obtained over 60 seconds are presented in Graph No. 10 below:



Graph 10

As can be seen, the quantification method applied to a very thin film correlated well with the drying rate profile of the ink.

Example 13

Using the same equipment and same process as in Example 12, an UV curable flexographic ink (FR Cyan FLHFV 5480055) and UV coating (R2964-156) were applied to the substrate. The changes in the ink film's resistance before and after curing were measured using a Fluke® 189 digital multimeter. The results are presented in Table 5 below.

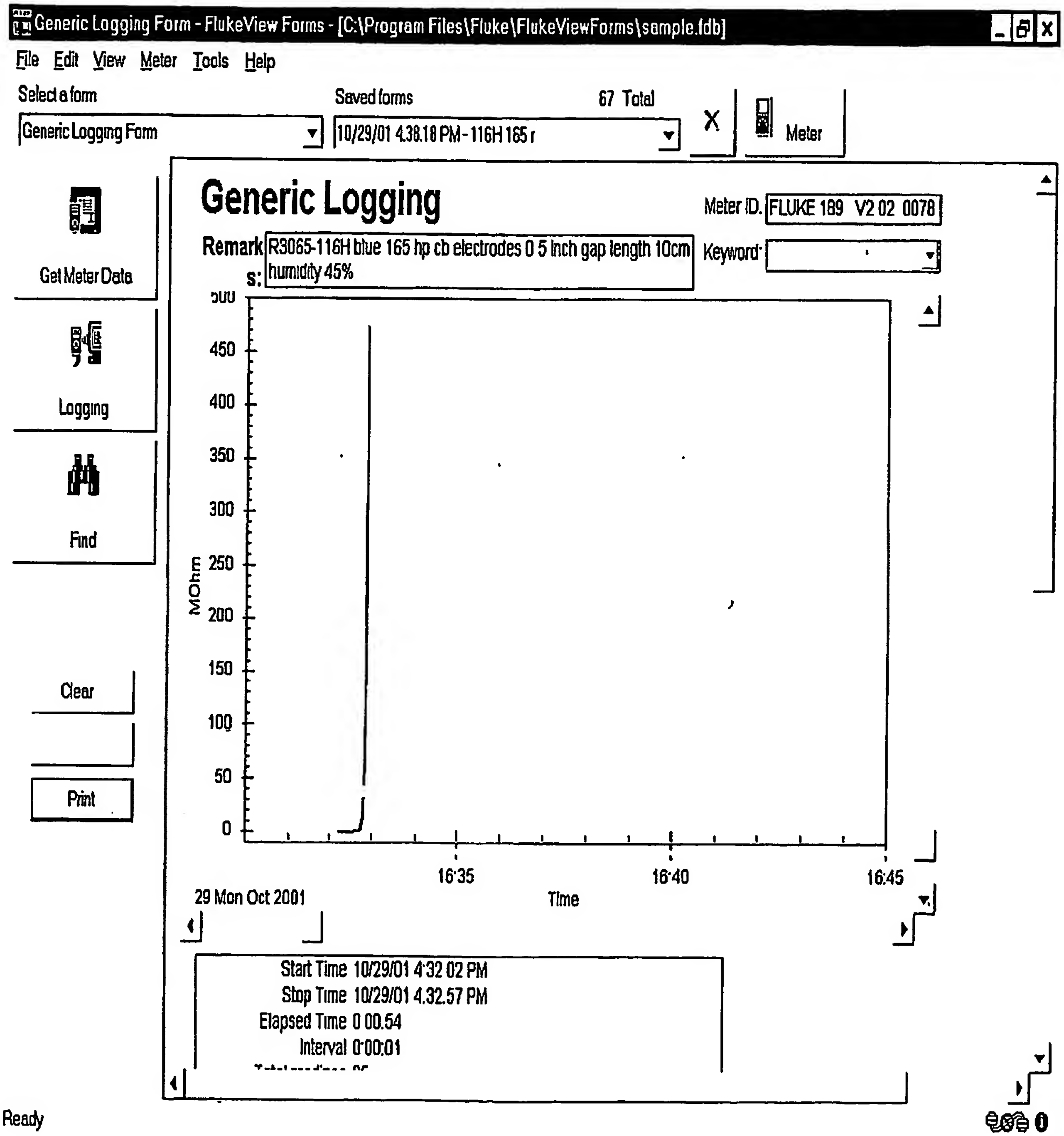
Table 5
Resistance Before and After Energy Curing

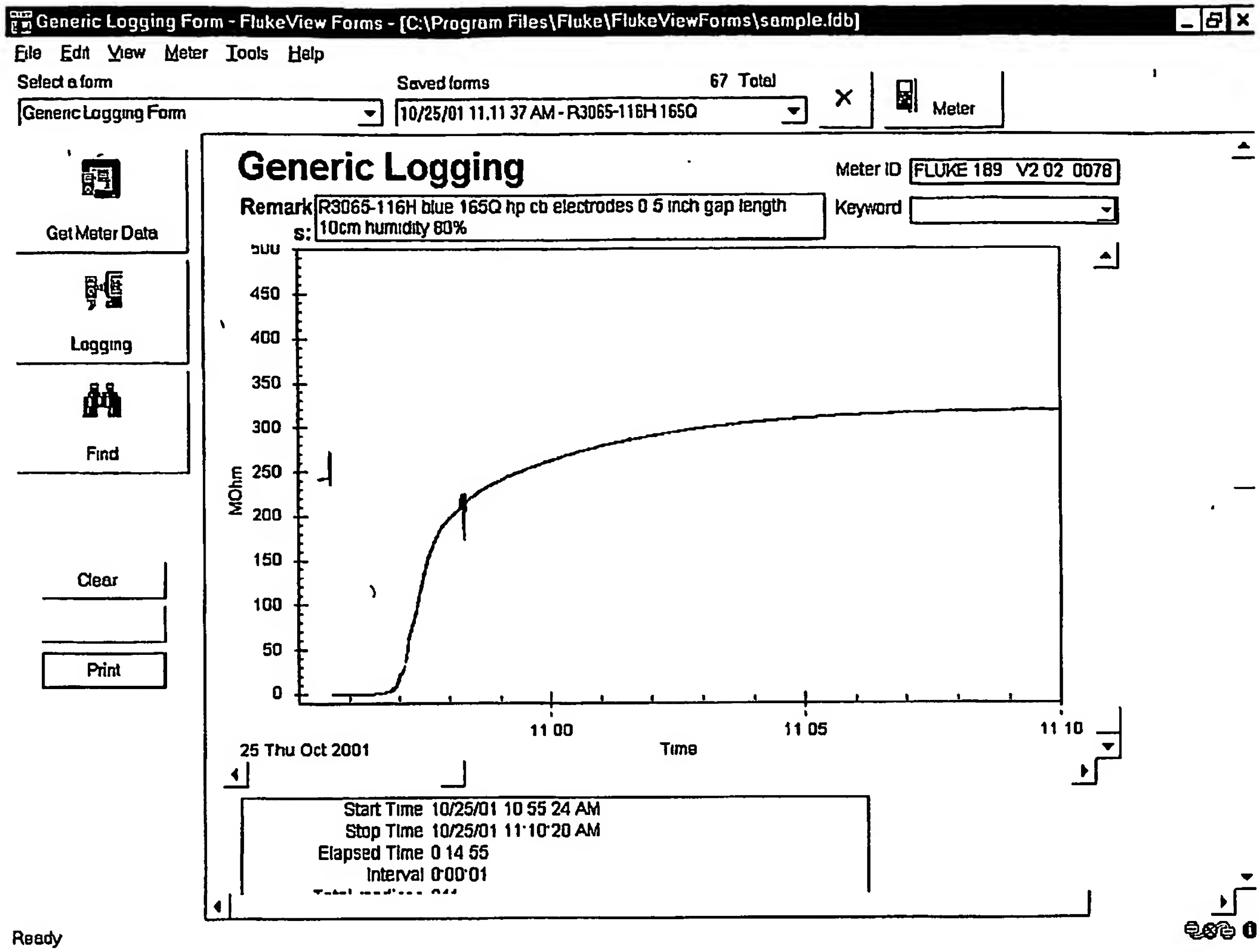
Sample	UV Flexo Ink	UV coating
Before curing	59 Mohms	359 Mohms
After Curing	5,000 Mohms	5,500 Mohms

Example 14

A water-based blue laminating ink (DPF - 426, available from Sun Chemical) was applied via a flexographic handproofer (165 lines per inch) onto a Lenata Board™ having 2 pairs of imprinted electrodes on the surface. The length of each pair of electrodes was 10 cm. and the space between the pair of electrodes was 1.25 cm. The measurements were performed at humidity levels of stabilized 45% and 80% humidity. Changes in ink film resistance were recorded with a Fluke® 189 digital

multimeter (having resistance range of 0-500 Mohm). The results obtained over a measurement period of 15 minutes are present below in Graphs 11 and 12.





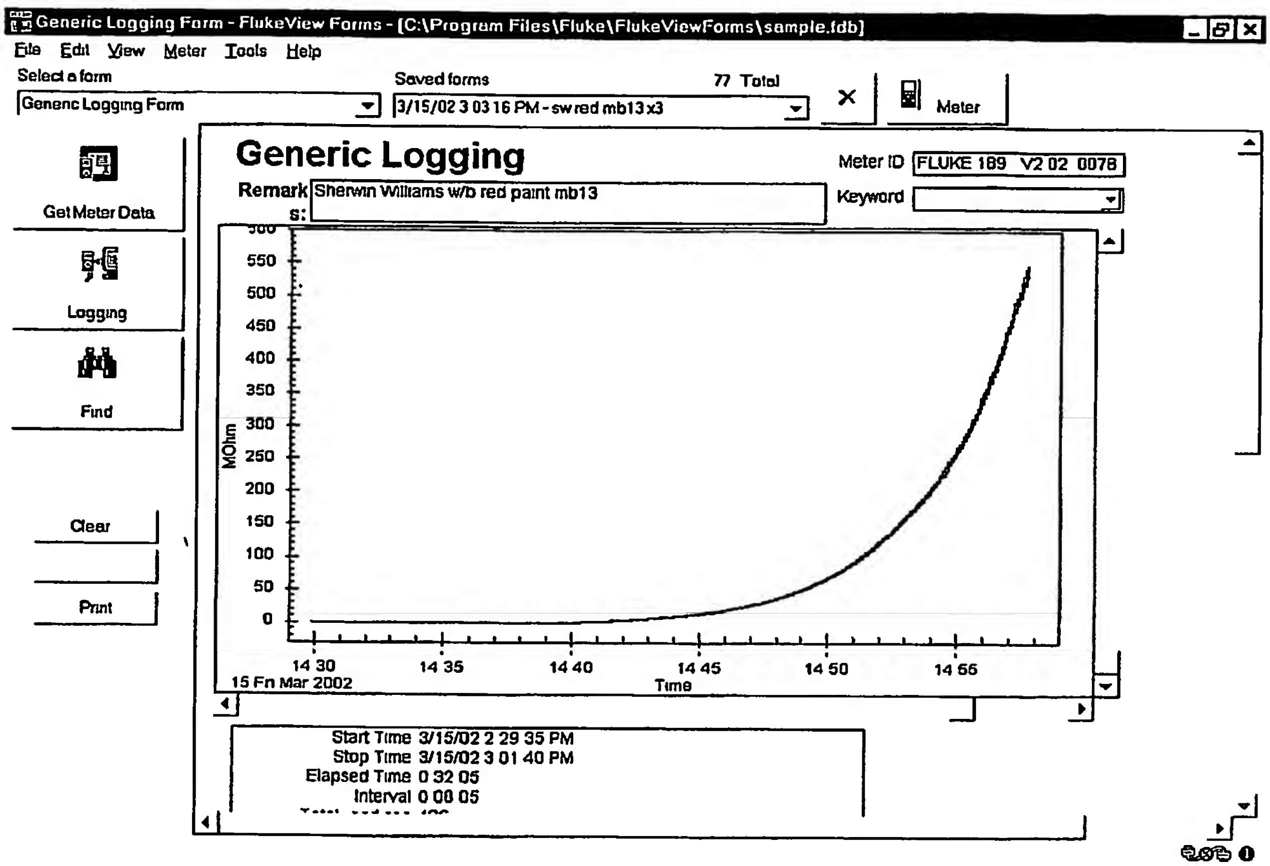
Graph 12

As can bee seen, the results confirm that water based ink film dry faster at low humidity.

Example 15

A water-based paint (available from Sherwin Williams DTA Acrylic Gloss Coating, Safety Red B66 R38 136-559) was applied via a

Mayer Bar #13 onto a Lenata Board™ having a pair of electrodes the same length and spaced apart the same distance as described in Example 15 above. Change in the ink film resistance was recorded using the same Fluke® 189 digital multimeter as described in Example 14 above. The results obtained over a 30 minute period are presented in Graph 13.

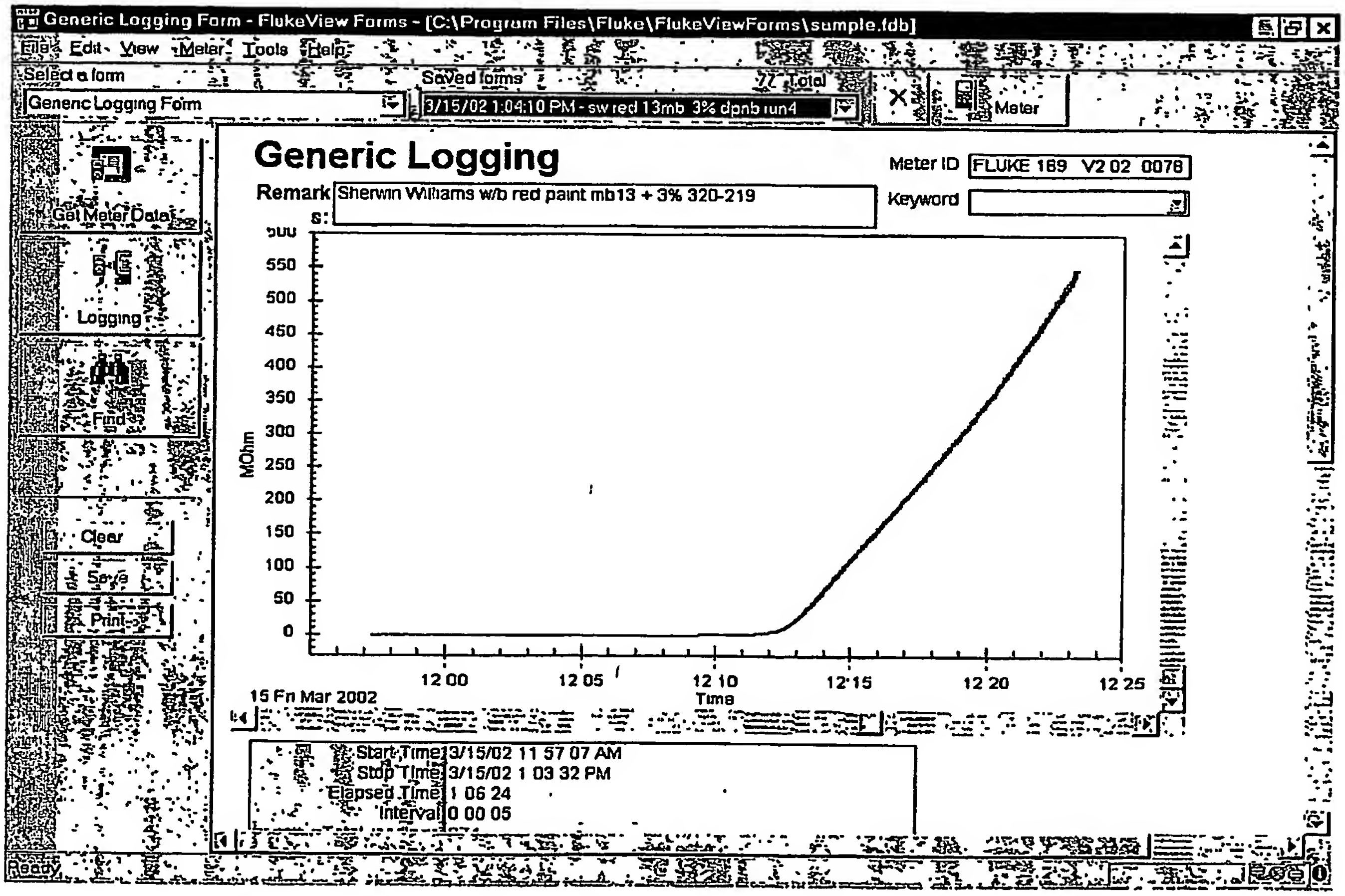


Graph 13

As can be seen, the results confirm that the method and apparatus can be used to profile the effect of additives on the drying rate of paints.

Example 16

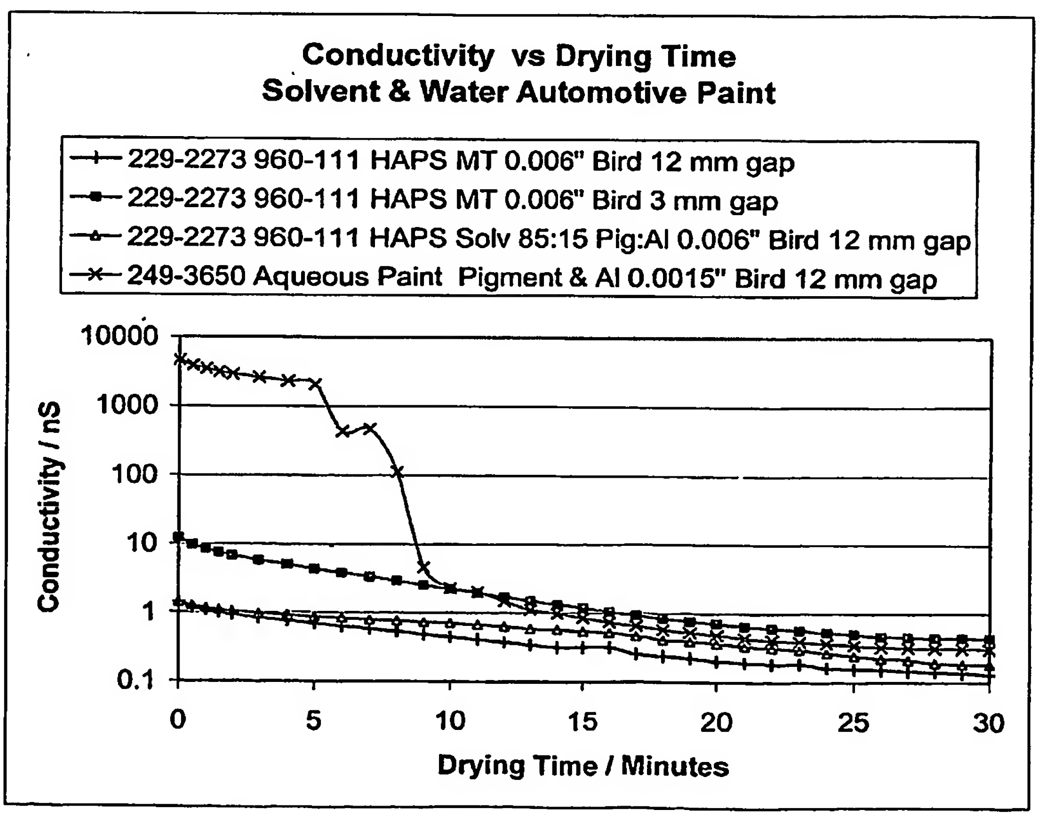
Diproplyene glycol (3%) was added to the water based paint of Example 16 and the modified paint was applied and tested via the same equipment and substrate as described in Example 16. Change in the ink film resistance was recorded over a 30 minute period and the results are presented in Graph 14 below:



As can be seen, the results confirm that the method and apparatus can be used to profile the effect of additives on the drying rate of paints.

Example 17

The drying rate profiles of two solvent and one water-based automotive paint systems were measured using the procedure described in Example 16. The solvent based (n-butyl propionate) paint systems, one containing aluminum flakes and one containing a water based paint system containing aluminum flakes, were tested. The three paints were each separately applied onto two different Leneta Boards, both having imprinted electrodes the same length as in Example 15 described above but with one pair of electrodes being spaced apart 1.25 cm. and the other pair of electrodes being spaced apart 3 mm. Change in the ink film resistance was record using the same Fluke 189® digital multimeter as in Example 15 described above. The results obtained over a 30 minute period are provided in Graph 15.



As can be seen, the results confirm that the drying rate profile f0or water based automotive systems is dramatically different from that for solvent-based automotive paint systems. The method and apparatus were successfully used to profile drying rate of automotive paints.

The foregoing examples are not intended to be limiting. Other examples and applications will be apparent to persons of skill in the art. The scope of the invention is established by the following set of claims.

Claims

What is claimed is:

1. A method for determining the drying rate of a liquid, comprising:
   - a.) applying an amount of the liquid onto a first piece of a substrate to form an initial rollout proof having a head where the liquid is first applied to the substrate and at least one tail where the liquid is last applied to the substrate;
   - b.) allowing the liquid to at least partially dry for a predetermined period of time;
   - c.) repeating steps (a) - (b) to prepare at least one second rollout proof on a second piece of the substrate, said after rollout proof having a head where the liquid is first applied to the substrate and at least one tail where the liquid is last applied to the substrate, and allowing the second rollout to at least partially dry for the same predetermined period of time allowed to elapse in (b);
   - d.) utilizing a densitometer to measure the density of each of the at least partially dried first and second rollout proofs, at the tails of the respective rollout proofs;
   - e.) repeating steps (a) - (d) a plurality of times,;
   - f.) plotting the measured density of the tails of each of the respective rollout proofs versus a parameter related to the elapsed time at which the density measurement was made, to form a graph with at least one curve which is representative of the drying rate of the liquid.

2. The method according to claim 1, wherein at least three replications of each sample are performed and average values of all of the measured density values for corresponding replications are plotted to form the graph.

3. The method according to claim 1, wherein in step (e), from 2 to 20 repetitions of steps (a) - (d) are performed until complete drying of the sample is attained, to provide a corresponding number of density measurements at the tails of the rollout proofs for use in plotting to form the graph of the curve representative of the drying rate of the liquid.

4. The method according to claim 1, wherein the liquid is selected from the group consisting of coatings, inks, and paints.

5. The method according to claim 1, wherein when the liquid is an ink.

6. The method according to claim 1, wherein when the liquid is a flexographic ink.

7. The method according to claim 6, wherein the flexographic ink is selected from the group consisting of: water-based ink for paper packaging, solvent-based ink for film, and water-based ink for film.

8. The method according to claim 1, wherein the at least one curve that is representative of the drying rate of the liquid is a plot of print density of the liquid, as measured by the densitometer at a predetermined point of time, versus sample

number or tail number corresponding to that densitometer measurement.

9. The method according to claim 10, wherein the at least one curve representative of the drying rate of the liquid is selected from a plot of: average initial and average after density measurements versus sample or tail number; differences between the average after and average initial density measurements ($\Delta = \rho_A - \rho_I$) versus sample or tail number; and differences between the difference between the initial and after density and a density of a reference base substance ($\Delta = [(\rho_A - \rho_I) - \rho_R]$) versus sample or tail number

10. The method according to claim 1, wherein step (f) is automated.

11. The method according to claim 11, wherein the automation of step (f) includes a computer system with computer graphics hardware and software which is connected to the densitometer, such that densitometer measurements of the density of the tails of rollout proofs are directly inputted to the computer and the graph of the curve is automatically plotted.

12. An apparatus for determining the drying rate of a liquid, comprising:

a.) container means for containing a volume of the liquid;

b.) a capillary tube, having a predetermined diameter, and first and second opposite open ends, such that a first end of the capillary tube is capable of being immersed in the volume of said liquid;

c.) an inert gas source for supplying a flow of an inert gas that is not chemically reactive with said liquid, said inert gas source being connected to said second open end of said capillary tube; and

d.) pressure-sensing means for measuring a pressure of said flow of said inert gas, and clock means for measuring a length of time, that is required to force an amount of said liquid, that has been drawn from said volume of said liquid in said container into said capillary tube through said first open end of said capillary tube by capillary action, after said first open end of said capillary tube has been immersed into said volume of said liquid, from said capillary tube and for a bubble of said inert gas to form in said container means containing said volume of said liquid whose drying rate is being measured.

13. An apparatus of claim 14 wherein a second capillary tube is included.

14. A method for determining the drying rate of a liquid, comprising:

a.) providing a container holding a volume of the liquid;

b.) providing a capillary tube, having a predetermined diameter, and first and second opposite open ends, such that said first end of said capillary tube is capable of being inserted into said container;

c.) providing a source for supplying an inert gas that is not reactive with said liquid whose drying rate is being determined, connected to said second open end of said capillary tube;

d.) immersing said first open end of said capillary tube into said container holding said volume of said liquid whose drying rate is being determined, so as to cause an amount of said liquid to be drawn into said first open end of said capillary tube by capillary action;

e.) causing an amount of said inert gas, of measured pressure, to flow into said second open end of said capillary tube, simultaneously with (d), and measuring a length of time until said amount of liquid in said capillary tube has been forced out therefrom and a bubble of said inert gas is detected in said container holding said volume of said liquid;

f.) repeating steps (d) and (e);and

g.) determining the drying rate of said liquid from measurements of the lengths of time and increases in pressure required, from one measurement to a subsequent measurement, of said inert gas that is required to force said liquid out from said first open end of said capillary tube and for said bubble of said inert gas to form in said container of said liquid.

15. An apparatus of claim 15 wherein a second capillary tube is included.

16. An apparatus for determining the electrical conductance of a liquid, comprising:

a.) an application means for applying said liquid onto a substrate; and

b.) at least one pair of electrodes on said substrate connected to a multimeter capable of reading electrical conductance and/or electrical resistance.

17. The apparatus according to claim 15 wherein said liquid if selected from the group consisting of: coatings, inks, paints, lacquers and adhesives.

18. The apparatus according to claim 16, wherein when the liquid is an ink.

19. The apparatus according to claim 16, wherein when the liquid is a flexographic ink.

20. The apparatus according to claim 18, wherein the flexographic ink is selected from the group consisting of: water-based ink for packaging, solvent-based ink for film, and water-based ink for film.

21. The apparatus according to claim 15, wherein when the liquid thickness ranges from 0.001mm to 5.0 mm.

22. The apparatus according to claim 15, wherein when the pair of electrodes is each 1 cm to about 30 cm in length.

23. The apparatus according to claim 15, wherein when one of the pair of electrodes is spaced between 0.1 mm to 200 mm from the other.

24. The apparatus according to claim 15, wherein the pair of electrodes is made of conductive metal.

25. The apparatus according to claim 25 wherein the conductive metal is aluminum foil.

ABSTRACT OF THE DISCLOSURE

Apparatus and method for determining the drying rate of liquid film in air or other gaseous media. An apparatus and method for determining the drying rate of liquid film by measuring changes in a density-related parameter of the liquid over time, as measured from the tails of rollout samples of the liquid are also disclosed. An apparatus and method for measuring the drying rate of a liquid as correlated to changes in the dynamic surface tension of the liquid over time, as measured by the pressure of an inert gas required to displace a sample of the liquid, drawn into a capillary tube from a reservoir of the liquid, and for a bubble of the gas to form in the reservoir of liquid, are disclosed. An apparatus and corresponding method for measuring the electrical conductance/resistance of a' liquid over time, and for correlating such measurements to drying rates of the liquid are also disclosed.

APPENDIX II

ROLLOUT OF FLEXOMAX 33 MAGENTA ON FILM

Circle represents where density readings are taken.

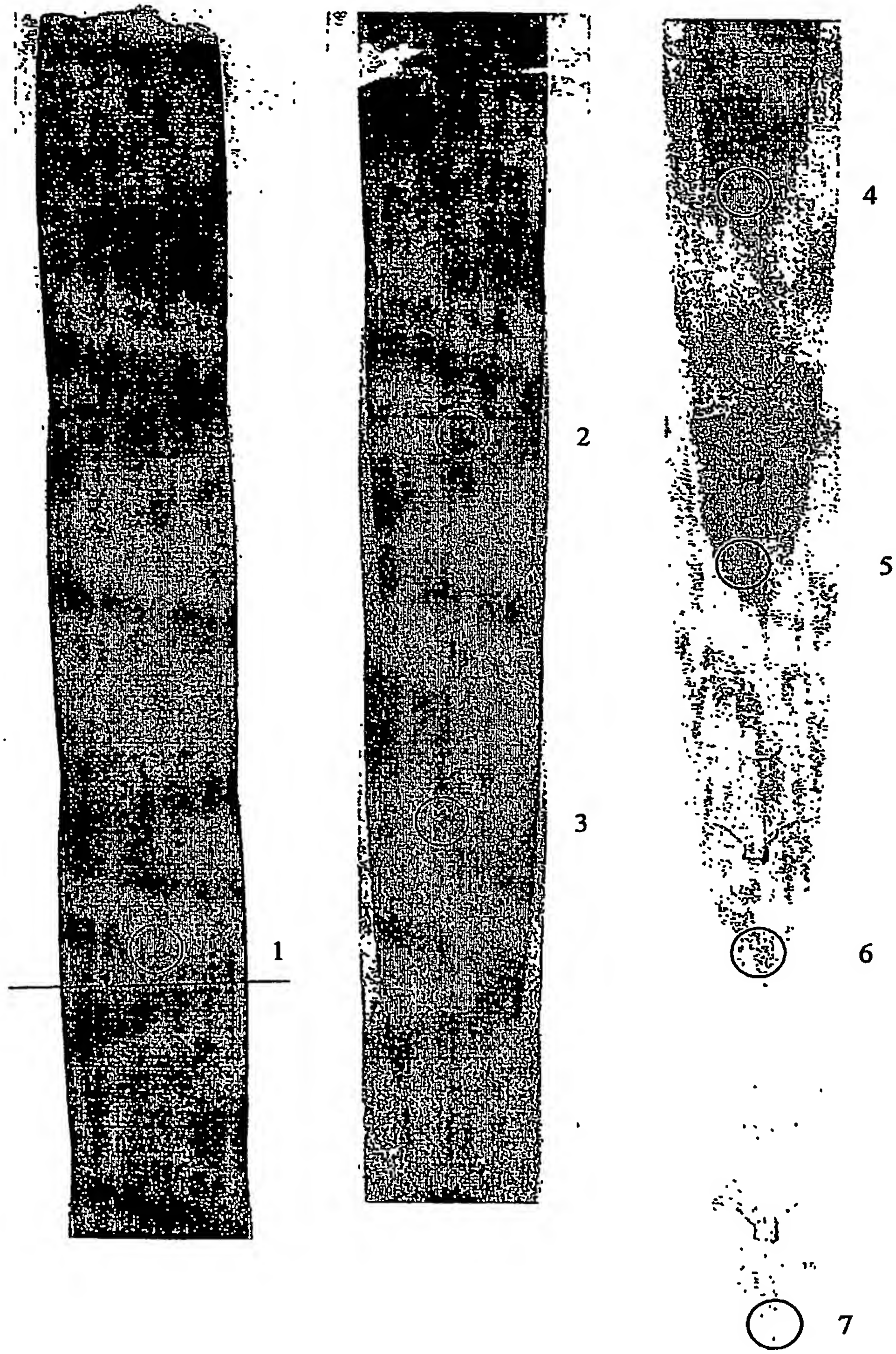


Fig. 1

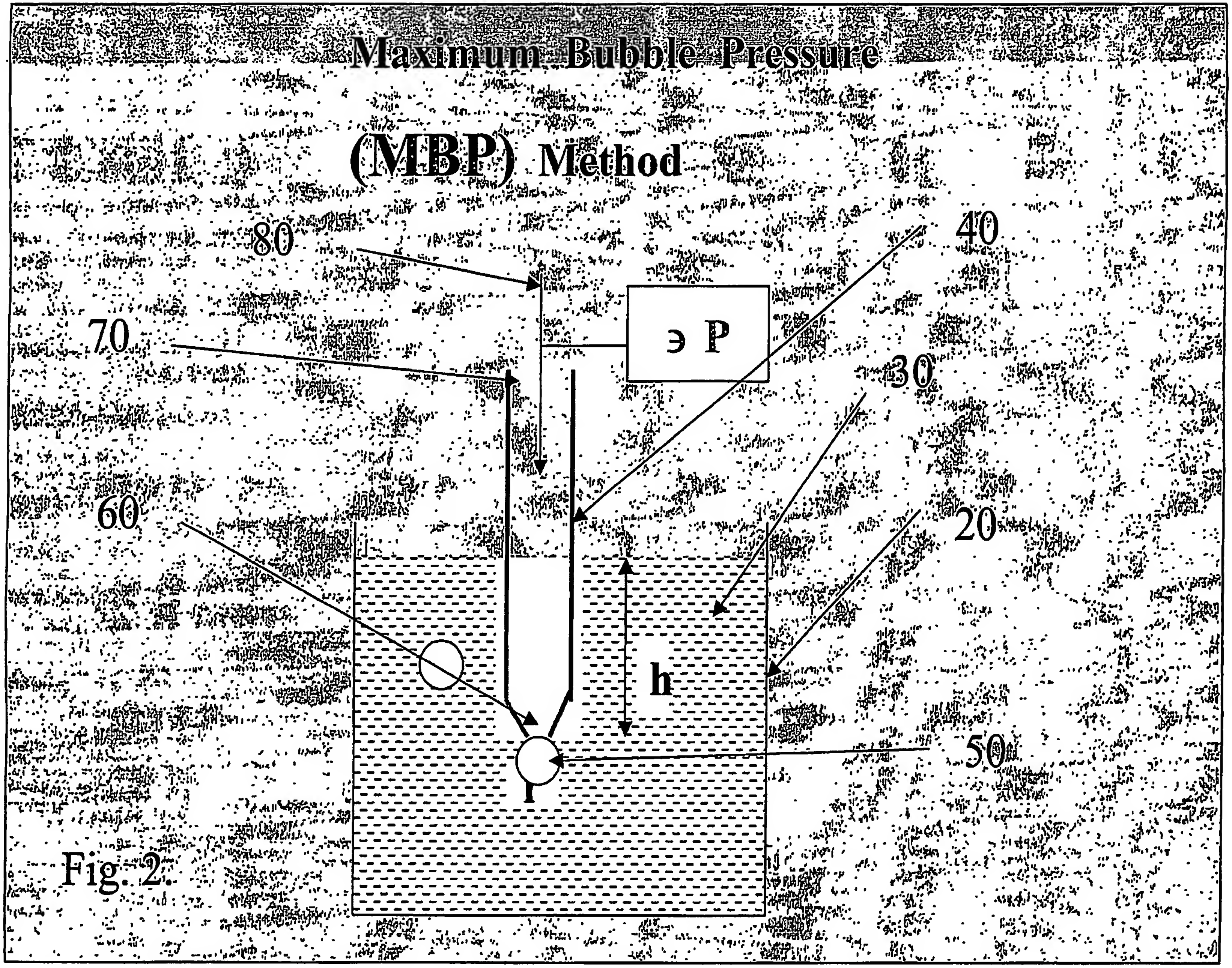


Fig. 2.

Set-up for determination of ink drying - schematic diagram

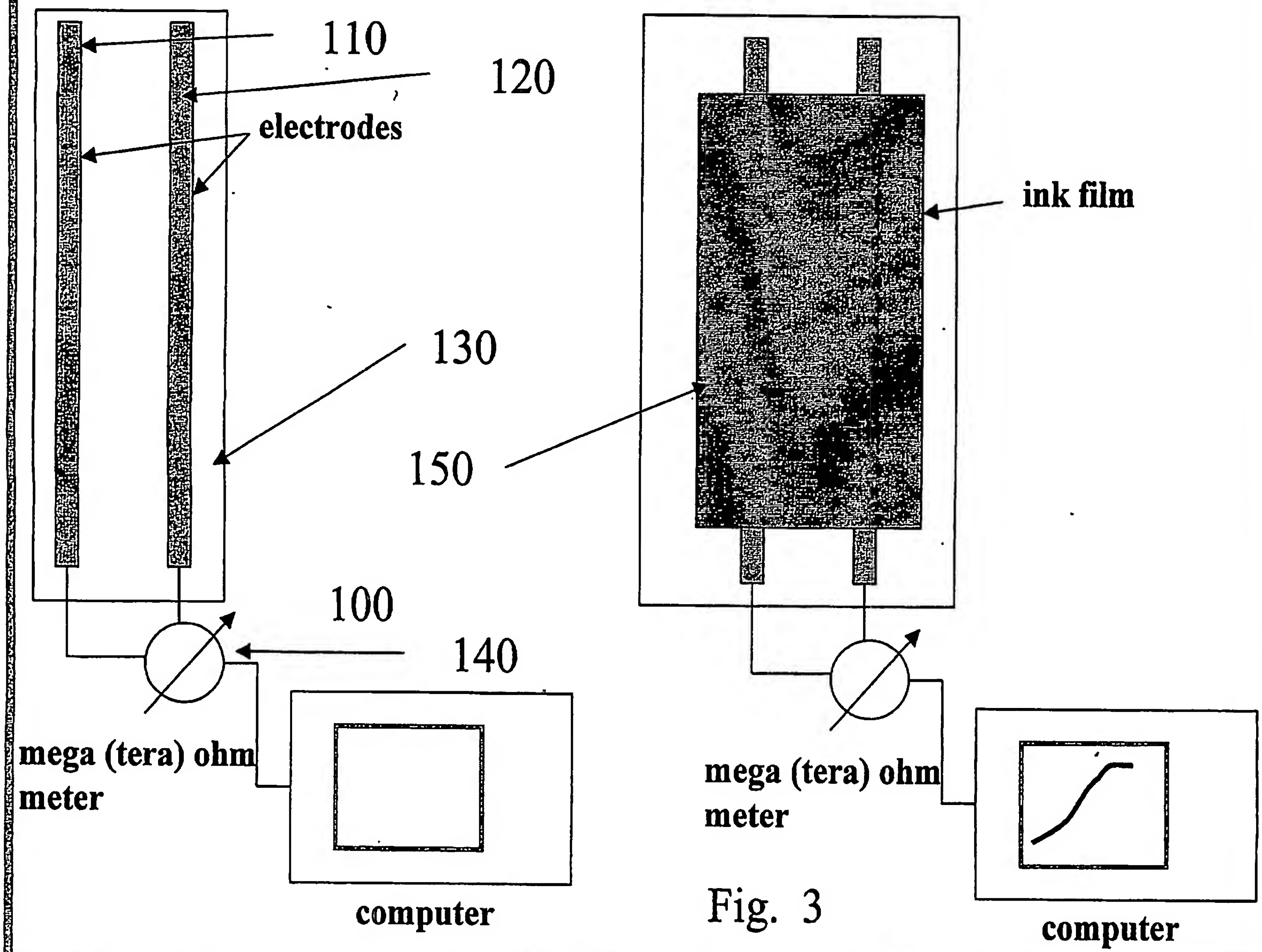


Fig. 3